

SYNTHESIS AND CHARACTERIZATION OF  
THERMORESPONSIVE POLY (N-  
VINYLCAPROLACTAM) / FILLERS  
NANOCOMPOSITE

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## **SUPERVISOR'S DECLARATION**

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy.

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I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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SYNTHESIS AND CHARACTERIZATION OF THERMORESPONSIVE POLY  
(N-VINYLCAPROLACTAM) / FILLERS NANOCOMPOSITE

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## ABSTRAK

Poly (N-vinylcaprolactam) (PNVCL) telah menarik perhatian banyak penyelidikan baru-baru ini sebagai salah satu polimer thermoresponsive menjanjikan. Walaupun terdapat satu faktor penting yang menjadikan PNVCL sangat menarik; ia telah dilaporkan bahawa PNVCL perlu meningkatkan ciri-ciri mekanikal, biocompatibility, dan macroporosity untuk menjadikannya calon yang menarik untuk aplikasi bioperubatan. Oleh itu, nanoteknologi telah digunakan sebagai satu cara untuk meningkatkan sifat-sifat PNVCL dengan menambah jumlah sedikit nanofiller samping memelihara ciri-ciri suhu yang responsif. Dalam kerja-kerja ini, parameter yang ketara pempolimeran telah mengenal pasti, kemudian melaksanakan mensintesis dan pencirian polimer thermoresponsive / pengisi nanocomposites. Satu siri nanocomposites PNVCL telah dibangunkan dan digabungkan dengan nanofillers (organo (C20) dan maghemite nanotube karbon multiwalled (Fe-MWCT)) melalui proses pempolimeran in-situ dibantu oleh kaca magnet. nanocomposites PNVCL yang disintesis tertakluk kepada proses pencirian yang berbeza seperti kestabilan haba, penukaran, perubahan morfologi, bengkak dan sifat-sifat reologi menggunakan kaedah pencirian yang berbeza seperti sinar-X pembelauan (XRD), Fourier Transform Infrared Spektroskopi (FTIR), Nuklear spektroskopi resonans magnet (NMR), dan Scanning Electron Microscopy (SEM). Kestabilan haba yang nanocomposites telah ditentukan dengan menggunakan analisis Termogravimetri (TGA) dan Differential Scanning Calorimetry (DSC). Daripada keputusan, yang diperhatikan rendah dan luas puncak XRD daripada nanocomposites disahkan rejim sudut yang lebih rendah daripada sampel disebabkan oleh pengembangan jarak basal. Peningkatan dalam kandungan tanah liat dan Fe-MWCNTs meningkat pengembangan d-jarak daripada nanocomposites. Selain itu, keputusan FTIR menunjukkan band penyerapan min kumpulan berfungsi utama dalam nanocomposites seperti hidrogen percuma karbonil (C = O), hidrogen terikat -OH regangan dan percuma -OH regangan. Selain itu, <sup>1</sup>H dan <sup>13</sup>C NMR telah digunakan untuk struktur polimer dan produk degradasi pencirian nanocomposites PNVCL. Keputusan TGA menunjukkan peningkatan yang ketara dalam nanocomposites PNVCL selepas penubuhan C20 dan Fe-MWCNTs. Keserasian Fe-MWCNTs dengan PNVCL didapati lebih tinggi berbanding dengan C20 dalam matriks polimer. Ini terbukti yang lebih tinggi d-jarak, haba, dan sifat-sifat mekanik Komposit nano yang dibentuk dengan 0.3% berat Fe-MWCNT berbanding Komposit nano dibentuk dengan 3% berat C20. Struktur diinterkalasi daripada nanocomposites PNVCL diberikan kestabilan haba yang baik kepada nanocomposites seperti yang ditentukan oleh TGA. Lengkung DTG menunjukkan tiada perbezaan yang signifikan kandungan C20 atau Fe-MWCNT lebih tinggi ke atas proses penyahkutuban, menunjukkan kesan positif daripada pengisi kepada proses degradasi terma. Reka bentuk pusat komposit (CCD) daripada kaedah gerak balas permukaan (RSM) telah digunakan semasa proses pengoptimuman dalam kajian ini. Proses pengoptimuman dilakukan dengan tiga faktor proses (suhu, masa, dan jumlah nanofillers) dan keputusan menunjukkan kenaikan suhu dan nanofillers kandungan untuk memihak kepada pempolimeran daripada nanocomposites ke tahap tertentu. Model kuadratik dibangunkan adalah agak tepat. Kekurangan yang tidak ketara kesilapan peratusan patut dan rendah semasa eksperimen pengesanan menunjukkan kesahihan proses pengoptimuman pada tahap yang penting bagi kedua-dua nanocomposites.

## ABSTRACT

Poly (N-vinylcaprolactam) (PNVCL) has attracted much research attention recently as one of the promising thermoresponsive polymers. Even there is an essential factor that makes PNVCL very attractive; it has been reported that PNVCL needs to improve its mechanical characteristics, biocompatibility, and macroporosity to make it an exciting candidate for biomedical applications. Therefore, nanotechnology has been used as a way to enhanced PNVCL properties by adding a little amount of nanofiller while preserving the temperature-responsive properties. In this work, the significant parameters of polymerization have been identifying, then perform the synthesize and characterization of thermoresponsive polymer/fillers nanocomposites. A series of PNVCL nanocomposites were developed and incorporated with nanofillers (organoclay (C20) and maghemite multiwalled carbon nanotubes (Fe-MWCNT)) via an in-situ polymerization process assisted by magnetic stirring. The synthesized PNVCL nanocomposites were subjected to different characterization processes such as thermal stability, conversion, morphology changes, swelling and rheological properties using different characterization methods like X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Nuclear magnetic resonance spectroscopy (NMR), and Scanning Electron Microscopy (SEM). The thermal stability of the nanocomposites was determined using Thermogravimetric analysis (TGA) and Differential Scanning Calorimetry (DSC). From the results, the observed low and broad XRD peaks of the nanocomposites confirmed the lower angle regime of the samples due to the expansion of the basal spacing. An increase in the clay and Fe-MWCNTs content increased the d-spacing expansion of the nanocomposites. Additionally, the FTIR results show the mean absorption bands of the main functional groups in the nanocomposites such as hydrogen free carbonyl (C=O), hydrogen-bonded -OH stretching and free -OH stretching. Moreover,  $^1\text{H}$  and  $^{13}\text{C}$  NMR was used for polymer structure and degradation products characterization of the PNVCL nanocomposites. The TGA results show a significant improvement in the PNVCL nanocomposites after the incorporation of C20 and Fe-MWCNTs. The compatibility of Fe-MWCNTs with PNVCL was found to be higher compared to that of C20 in the polymer matrix. This was evidenced in the higher d-spacing, thermal, and mechanical properties of the nanocomposite formed with 0.3 wt% Fe-MWCNT compared to nanocomposite formed with 3 wt% C20. The intercalated structure of the PNVCL nanocomposites conferred improved thermal stability to the nanocomposites as determined by TGA. The DTG curves show no significant influence of higher C20 or Fe-MWCNT contents on the depolarization process, indicating a positive effect of the fillers on the thermal degradation process. The central composite design (CCD) of the response surface methodology (RSM) was employed during the optimization process in this study. The optimization process was performed with three process factors (temperature, time, and the amount of nanofillers) and the results show increases in the temperature and nanofillers content to favor the polymerization of the nanocomposites to a certain extent. The quadratic model developed was reasonably accurate. The insignificant lack of fit and low percentage errors during the validation experiment showed the validity of the optimization processes at a significant level for both nanocomposites.

## TABLE OF CONTENT

<b>DECLARATION</b>	
<b>TITLE PAGE</b>	
<b>ACKNOWLEDGEMENTS</b>	<b>ii</b>
<b>ABSTRAK</b>	<b>iii</b>
<b>ABSTRACT</b>	<b>iv</b>
<b>TABLE OF CONTENT</b>	<b>v</b>
<b>LIST OF TABLES</b>	<b>x</b>
<b>LIST OF FIGURES</b>	<b>xiii</b>
<b>LIST OF SYMBOLS</b>	<b>xviii</b>
<b>LIST OF ABBREVIATIONS</b>	<b>xix</b>
<b>CHAPTER 1 INTRODUCTION</b>	<b>1</b>
1.1 Background	1
1.2 Motivation	2
1.3 Problem Statement	3
1.4 Objectives	5
1.5 Scope	5
1.6 The significance of the study	6
1.7 Thesis Outline	6
<b>CHAPTER 2 LITERATURE REVIEW</b>	<b>8</b>
2.1 Introduction	8
2.2 Thermoresponsive Polymer	8



2.3	Poly (N-vinylcaprolactam) (PNVCL)	10
2.3.1	Thermosensitivity	10
2.3.2	Synthesis of PNVCL	12
2.4	Polymer Nanocomposite	14
2.4.1	Structure of Polymer Nanocomposites	16
2.4.2	Developing of Polymer Nanocomposite	17
2.4.3	Methods of Preparing Polymer Nanocomposites	18
2.5	Nanofillers	21
2.5.1	Clay and Organoclay	22
2.5.2	Carbon Nanotubes (CNTs)	26
2.6	Characterization of Polymer/Fillers Nanocomposites	36
2.6.1	Structure analysis	36
2.6.2	Thermal Analysis	38
2.6.3	Rheological analysis	39
2.7	Applications	40
2.7.1	Poly (N -vinylcaprolactam) Applications	40
2.7.2	Polymer/ Organoclay Nanocomposite Applications	47
2.7.3	Magnetic-MWCNTs Nanocomposite Applications	50
2.8	Optimization	52
2.9	Summary	55
<b>CHAPTER 3 METHODOLOGY</b>		<b>57</b>
3.1	Introduction	57
3.2	Materials	57
3.3	Methodology Framework	58
3.3.1	Polymerization process	58

3.3.2	Experimental Setup	58
3.4	Synthesis Process	60
3.4.1	Synthesis of Poly (N-vinylcaprolactam) PNVCL/Organoclay Nanocomposite.	60
3.4.2	Synthesis of Poly (N-vinylcaprolactam) PNVCL/Maghemite– Multiwalled Carbon Nanotube (Fe-MWCNT) Nanocomposite.	62
3.5	Characterization	65
3.5.1	Structure Analysis	65
3.5.2	Thermal Analysis	67
3.5.3	Rheology Analysis	68
3.5.4	Swelling Analysis	69
3.6	Screening and Optimization Analysis for Synthesis of polymer/organoclay nanocomposite.	69
3.6.1	Screening design	70
3.6.2	Optimization Design	71
3.7	Summary	71
 <b>CHAPTER 4 RESULTS AND DISCUSSION</b>		 <b>73</b>
4.1	Introduction	73
4.2	Screening Analysis of Poly (N-vinylcaprolactam) (PNVCL) Nanocomposite	74
4.3	Characterization of PNVCL Based on Cloisite C20 Nanocomposites	82
4.3.1	Conversion of PNVCL Based on Cloisite C20 Nanocomposites	82
4.3.2	Structure analysis of PNVCL/C20 Nanocomposites	84
4.3.3	Thermal Analysis of PNVCL/C20 Nanocomposites.	94
4.3.4	Swelling Analysis of PNVCL/C20 Nanocomposites.	99

4.4	Characterization of PNVCL Based on Fe-MWCNTs Nanocomposites	101
4.4.1	Conversion of PNVCL-Based on Fe-MWCNT Nanocomposites	101
4.4.2	Structure Analysis of PNVCL/Fe-MWCNTs Nanocomposites.	103
4.4.3	Thermal Analysis of PNVCL/Fe-MWCNTs Nanocomposites.	110
4.4.4	Swelling Analysis of PNVCL/Fe-MWCNTs Nanocomposites.	115
4.5	Comparative Study of PNVCL, PNVCL/Organoclay (C20) and PNVCL/ Maghemite-Multiwalled Carbon Nanotubes (Fe-MWCNTs) Nanocomposites.	117
4.5.1	Fourier Transform Infrared Spectroscopy (FTIR) of PNVCL, PNVCL/C20 and PNVCL/Fe-MWCNTs Nanocomposites.	117
4.5.2	X-ray diffraction (XRD) of PNVCL, PNVCL/C20 and PNVCL/Fe-MWCNTs Nanocomposites.	118
4.5.3	Ultraviolet-visible spectroscopy (UV-Vis) of PNVCL, PNVCL/C20 and PNVCL/Fe-MWCNTs Nanocomposites.	119
4.5.4	Scanning electron microscope (SEM) of PNVCL, PNVCL/C20 and PNVCL/Fe-MWCNTs Nanocomposites.	120
4.5.5	Nuclear magnetic resonance spectroscopy ( $^1\text{H}$ and $^{13}\text{C}$ - NMR) of PNVCL, PNVCL/C20 and PNVCL/Fe-MWCNTs Nanocomposites.	122
4.5.6	Thermogravimetric analysis (TGA) of PNVCL, PNVCL/C20 and PNVCL/Fe-MWCNTs Nanocomposites.	126
4.5.7	Differential scanning calorimetry (DSC) of PNVCL, PNVCL/C20 and PNVCL/Fe-MWCNTs Nanocomposites.	127
4.5.8	Rheological Analysis of PNVCL, PNVCL/C20 and PNVCL/Fe-MWCNTs Nanocomposites.	129
4.6	Optimization of Poly (N-vinylcaprolactam) (PNVCL) Nanocomposite using Response Surface Methodology (RSM).	132
4.6.1	Optimisation of PNVCL/C20 Nanocomposite	132

4.6.2	Validation of the PNVCL/C20 Nanocomposite	144
4.6.3	Optimisation of PNVCL/Fe-MWCNTs Nanocomposites	144
4.6.4	Validation of the PNVCL/Fe-MWCNTs Nanocomposite	154
4.7	Summary	155
<b>CHAPTER 5 CONCLUSION</b>		<b>157</b>
5.1	Conclusions	157
5.2	Recommendations for Future Research	159
<b>REFERENCES</b>		<b>161</b>
<b>APPENDIX A PUBLICATIONS</b>		<b>197</b>

## LIST OF TABLES

Table 2.1	Comparison between PNIPAM and PNVCL	11
Table 2.2	Carbon nanotubes CNTs /polymer by “grafting to” method	29
Table 2.2	Continued	30
Table 2.3	Carbon nanotubes CNTs /polymer by “grafting form” method	33
Table 2.3	Continued.	34
Table 2.3	Continued.	35
Table 2.4	Summary of thermoresponsive poly (N-vinylcaprolactam) (PNVCL) application	40
Table 2.4	Continued	41
Table 2.5	Compendium of the novel substantial studies on the poly (N-vinyl caprolactam) PNVCL for Pharmaceutical and biomedical applications.	43
Table 2.5	Continued.	44
Table 2.6	Polymer/clay nanocomposites applications	49
Table 2.7	Magnetic-MWCNTs Nanocomposite Applications.	51
Table 3.1	Chemicals and materials utilized for the synthesis process.	57
Table 3.1	Continued.	58
Table 3.2	Composition of the PNVCL/C20 nanocomposite.	61
Table 3.3	Composition of the PNVCL/Fe-MWCNT nanocomposite.	64
Table 3.4	Signifies the experimental results of the synthetic parameters at numerous variation arrangements.	70
Table 3.5	Experimental run for screening analysis	71
Table 4.1	Experimental design and results for polymer/ organoclay (C20) polymerization.	74
Table 4.2	Analysis of Variance for Conversion ratio (Y %).	75
Table 4.3	Estimated Regression Coefficients for Conversion%.	76
Table 4.4	Peak assignments for the FT-IR spectrum of NVCL and PNVCL	85
Table 4.5	The characteristic bands in the IR- spectra of Cloisite C20	86
Table 4.6	The characteristic bands in the IR spectra of PNVCL/C20 nanocomposites	88
Table 4.7	XRD data of C20, PNVCL and PNVCL/C20 nanocomposites for d-spacing and $2\theta$ .	91
Table 4.8	Crystallinity of PNVCL and PNVCL/C20 nanocomposites	92
Table 4.9	DTG-determined maximum temperature from PNVCL and PNVCL/C20 nanocomposites.	96

Table 4.10	TGA-determined weight losses and residue amounts from PNVCL and PNVCL/C20 nanocomposites at 700 °C.	97
Table 4.11	TGA-determined weight losses and residue amounts from PNVCL and PNVCL/C20 nanocomposites at different temperatures.	99
Table 4.12	The characteristic bands in the IR spectra of Fe-MWCNTs.	104
Table 4.13	The characteristic bands in the IR spectra of PNVCL /Fe-MWCNTs nanocomposites	105
Table 4.14	Crystallinity of PNVCL and PNVCL/ Fe-MWCNTs nanocomposites	108
Table 4.15	TGA data for PNVCL and PNVCL/Fe-MWCNT nanocomposites under nitrogen flow.	112
Table 4.16	DTG-determined maximum temperature from PNVCL and PNVCL/Fe-MWCNTs nanocomposites.	113
Table 4.17	Decomposition and variation temperatures for different weight percentages of PNVCL/Fe-MWCNTs nanocomposites.	113
Table 4.18	TGA-determined weight losses and residue amounts from PNVCL and PNVCL/ Fe-MWCNTs nanocomposites at different temperatures.	115
Table 4.19	XRD data for pure PNVCL, PNVCL/C20, and PNVCL/Fe-MWCNTs nanocomposites.	119
Table 4.20	TGA weight loss temperatures and residues of PNVCL, PNVCL/C20 with 3 wt% C20, and PNVCL/Fe-MWCNT with 0.3% nanocomposites.	127
Table 4.21	Signifies the experimental results.	132
Table 4.22	Experimental design for the optimization of the conversion ratio.	133
Table 4.23	Coefficients in Terms of Coded Factors.	134
Table 4.24	Analysis of variance (ANOVA) for response surface model of conversion ratio.	135
Table 4.25	The range of input parameters and response.	143
Table 4.26	Recommend optimum parameters for maximum conversion ratio of PNVCL/C20 nanocomposite.	144
Table 4.27	Results of validation experiment conducted at optimum combination of the PNVCL/C20 nanocomposite.	144
Table 4.28	signifies the experimental results.	145
Table 4.29	Experimental design for the optimization of the conversion ratio for PNVCL/Fe-MWCNT nanocomposite.	145
Table 4.30	Coefficients in Terms of C oded Factors.	146

Table 4.31	Analysis of variance (ANOVA) for response surface model of conversion ratio.	147
Table 4.32	The range of input parameters and response.	154
Table 4.33	Recommend optimum parameters for maximum conversion ratio.	155
Table 4.34	Results of validation experiment conducted at optimum combination of the PNVCL/Fe-MWCNT nanocomposite.	155

## LIST OF FIGURES

Figure 2.1	UCST and LCST referring the two graphical performances of phase diagrams (T is temperature, $\phi$ is the weight fraction of polymer in solution)	9
Figure 2.2	Structure of NVCL monomer and PNVCL polymer	10
Figure 2.3	RAFT polymerization of PNVCL	14
Figure 2.4	Scheme of different types of composites arising from the interaction of layered silicate and polymers: (a) phase separated microcomposite, (b) intercalated nanocomposite, (c) exfoliated nanocomposite.	16
Figure 2.5	Intercalation of polymer from solution.	18
Figure 2.6	Melt intercalation synthesis of polymer.	19
Figure 2.7	In-situ intercalative polymerization.	20
Figure 2.8	Structure of montmorillonite (MMT).	24
Figure 2.9	Different representations of clay dispersion with respect to the polymer via XRD analysis: A) pure clay, B) intercalated clay, C) exfoliated clay.	37
Figure 2.10	The plausible thermoresponsive released mechanism above LCST for the multi drugs from fib-graft-PNVCL NGs.	46
Figure 3.1	Overall methodology flowchart.	59
Figure 3.2	Experimental rig for the preparation of polymers.	60
Figure 3.3	Block diagram for the synthesis PNVCL/C20 nanocomposite.	61
Figure 3.4	Sample of PNVCL/C20 nanocomposite at 3 wt% organoclay (C20) ratio.	62
Figure 3.5	Schematic diagram for the synthesis Fe-MWCNTs.	63
Figure 3.6	Block diagram for the synthesis of PNVCL/Fe-MWCNT nanocomposite.	64
Figure 3.7	Sample PNVCL/Fe-MWCNTs nanocomposite at 0.4 wt%.	65
Figure 4.1	Residual Plots for Conversion ratio %.	76
Figure 4.2	Contour plot of (a): conversion (%) Vs organoclay % , monomer (g). (b): conversion (%) Vs organoclay % , initiaor (ml).	77
Figure 4.3	Contour plot of (a): conversion (%) Vs organoclay % , Temperature ( $^{\circ}$ C), (b): conversion (%) Vs organoclay % , Time (h).	78
Figure 4.4	Contour plot of (a): conversion (%) Vs temperature ( $^{\circ}$ C), monomer (g). (b): conversion (%) Vs temperature ( $^{\circ}$ C), initiaor (ml).	79



Figure 4.5	Contour plot of (a): conversion (%) Vs time (hr), monomer (g). (b): conversion (%) Vs time (h), initiaor (ml).	80
Figure 4.6	Pareto Chart of the Standardized Effect (Response is Conversion %, $\alpha = 0.05$ ).	81
Figure 4.7	Normal plot of the Standardized Effect (Response is Conversion %, $\alpha = 0.05$ ).	81
Figure 4.8	Conversion ratio against the organoclay (C20) ratio under the effect of temperature at different resident times: a) 4 h, b) 6 h, and c) 12 h.	83
Figure 4.9	FTIR Spectrum of monomer NVCL and polymer PNVCL.	84
Figure 4.10	FTIR spectra of organoclay (C20) and PNVCL.	86
Figure 4.11	FTIR spectra of PNVCL and PNVCL/organoclay nanocomposites with different percentages of organoclay (C20).	87
Figure 4.12	Comparative FTIR spectra of PNVCL and PNVCL/C20 nanocomposites (1-5 wt% organoclay) at a wavelength range of 1200 to 1800 cm <sup>-1</sup> .	88
Figure 4.13	Schematic of interfacial interaction of the PNVCL/Organoclay nanocomposite.	89
Figure 4.14	XRD patterns of PNVCL and purified organoclay C20.	89
Figure 4.15	Powder XRD patterns of organoclay (C20), PNVCL and PNVCL with different (1-5 wt %) organoclay contents.	90
Figure 4.16	UV-Vis absorbance spectra of PNVCL and PNVCL/C20 with different (1-5 wt%) organoclay contents.	93
Figure 4.17	UV-Vis transmittance spectra of PNVCL and PNVCL/C20 with different (1-5 wt%) organoclay contents.	93
Figure 4.18	TGA and DTG curves of PNVCL and PNVCL/C20 nanocomposites.	95
Figure 4.19	Differential scanning calorimetric curves of PNVCL and PNVCL/C20 nanocomposites.	99
Figure 4.20	The relationship between swelling ratio and time for the PNVCL and PNVCL/C20 nanocomposites.	100
Figure 4.21	Effect of organoclay (C20) content in the PNVCL nanocomposite on the swelling ratio at equilibrium state.	101
Figure 4.22	Conversion ratio against the Fe-MWCNT ratio under the influence of time and temperature.	102
Figure 4.23	FTIR spectra of Fe-MWCNTs and PNVCL.	103
Figure 4.24	FTIR spectra of PNVCL and PNVCL/Fe-MWCNTs nanocomposite with different percentages of Fe-MWCNTs.	104
Figure 4.25	FTIR spectra of PNVCL/Fe-MWCNTs nanocomposites with different percentages of Fe-MWCNTs.	105

Figure 4.26	Schematic of interfacial interaction of the PNVCL/Fe-MWCNTs nanocomposite.	106
Figure 4.27	XRD patterns of PNVCL and the purified Fe-MWCNTs.	107
Figure 4.28	Powder X-ray diffraction patterns of Fe-MWCNT and PNVCL/Fe-MWCNTs nanocomposites with various Fe-MWCNTs loadings.	107
Figure 4.29	UV-Vis absorbance spectra of PNVCL and PNVCL/Fe-MWCNTs nanocomposites.	109
Figure 4.30	UV-Vis transmittance spectra of PNVCL and PNVCL/Fe-MWCNTs nanocomposites.	110
Figure 4.31	TGA and DTG curves of PNVCL and PNVCL/Fe-MWCNT nanocomposites.	111
Figure 4.32	Differential scanning calorimetric (DSC) curves of PNVCL and PNVCL/Fe-MWCNT nanocomposites.	114
Figure 4.33	The relationship between swelling ratio and time for the PNVCL and PNVCL/ Fe-MWCNTs nanocomposites.	115
Figure 4.34	Effect of Fe-MWCNTs content in the PNVCL nanocomposite on the swelling ratio at the equilibrium state.	116
Figure 4.35	FTIR spectra of pure PNVCL, PNVCL/C20, and PNVCL/Fe- MWCNTs nanocomposites.	118
Figure 4.36	XRD plots of pure PNVCL, PNVCL/C20 nanocomposite, and PNVCL/Fe-MWCNTs nanocomposite.	119
Figure 4.37	UV-Vis spectra of pure PNVCL, PNVCL/C20 nanocomposite, and PNVCL/Fe-MWCNTs nanocomposite.	120
Figure 4.38	SEM images of the surface of a) pure PNVCL, b) PNVCL with 3 wt% C20 nanocomposite, and c) PNVCL with 0.3 wt% Fe-MWCNTs nanocomposite.	121
Figure 4.39	<sup>1</sup> H-NMR spectra of a) pure PNVCL, b) PNVCL/C20 nanocomposite at 3wt% and c) PNVCL/Fe-MWCNTs nanocomposite at 0.3 wt%.	123
Figure 4.40	<sup>13</sup> C-NMR spectra of a) pure PNVCL, b) PNVCL/C20 nanocomposite at 3wt% and c) PNVCL/Fe-MWCNTs nanocomposite at 0.3 wt%.	125
Figure 4.41	TGA curves for PNVCL, PNVCL/C20 and PNVCL/Fe-MWCNT nanocomposites .	126
Figure 4.42	Differential scanning calorimetric curves of PNVCL, PNVCL/C20, and PNVCL/Fe-MWCNT nanocomposites.	128
Figure 4.43	Differential scanning calorimetric LCST curves of PNVCL, PNVCL/C20, and PNVCL/Fe-MWCNT nanocomposites.	129

Figure 4.44	A representative graph of the storage modulus ( $G'$ ) and loss modulus ( $G''$ ) for a) PNVCL, b) PNVCL/3wt% C20, and c) PNVCL/0.3wt% Fe-MWCNTs nanocomposite as a function of temperature.	130
Figure 4.45	Dependence of complex viscosity on the shear rate for PNVCL, PNVCL/3wt% C20, and PNVCL/0.3 Fe-MWCNTs nanocomposites.	131
Figure 4.46	Normal probability plot of residual for conversion ratio of PNVCL/C20.	136
Figure 4.47	Plot of residuals versus predicted conversion ratio of PNVCL/C20.	137
Figure 4.48	Plot of residuals versus run number for the conversion ratio of PNVCL/C20.	137
Figure 4.49	Comparison between predicted and actual experiment values for the conversion ratio of PNVCL/C20.	138
Figure 4.50	Main plots of the conversion ratio (Y%) versus (A) time (B) temperature (C) C20 content.	139
Figure 4.51	Interaction between time, temperature and C20 content against conversion ratio of PNVCL/C20.	140
Figure 4.52	Counter and 3D surface plots of conversion ratio of PNVCL/C20 against of time and temperature.	141
Figure 4.53	Counter and 3D surface plots of conversion ratio of PNVCL/C20 against of time and C20 content.	142
Figure 4.54	Counter and 3D surface plots of conversion ratio of PNVCL/C20 against of temperature and C20 content.	143
Figure 4.55	Normal probability plot of residual for conversion ratio of PNVCL/Fe-MWCNT.	147
Figure 4.56	plot of residuals versus predicted conversion ratio of PNVCL/Fe-MWCNT.	148
Figure 4.57	plot of residuals versus run number for the conversion ratio of PNVCL/ Fe-MWCNT.	149
Figure 4.58	Comparison between predicted and actual experiment values for the conversion ratio of PNVCL/Fe-MWCNT.	149
Figure 4.59	One factor plot of conversion ratio as a function of (a) time (b) temperature (c) Fe-MWCNT content.	150
Figure 4.60	Interaction between time, temperature and Fe-MWCNT content against conversion ratio of PNVCL/Fe-MWCNT.	151
Figure 4.61	Counter and three-dimensional (3D) surface plots of conversion ratio of PNVCL/Fe-MWCNT against of time and temperature.	152

Figure 4.62	Counter and three-dimensional (3D) surface plots of conversion ratio of PNVCL/Fe-MWCNT against of time and Fe-MWCNT content.	153
Figure 4.63	Counter and three-dimensional (3D) surface plots of conversion ratio PNVCL/Fe-MWCNT against of temperature and Fe-MWCNT content.	153

## LIST OF SYMBOLS

Å	Angstrom
d	inter-clay layer space
g	Gram
G'	Storage modulus
G''	Loss modulus
hr	Hour
I	Initiator
L	Liter
m	Metre
M	Monomer
min	Minute
°	Degree
°C	Degree Celsius
P	probability values
R <sup>2</sup>	Coefficient of regression
T	Temperature
t	Time
T%	transmittance
T <sub>g</sub>	Glass transition temperature
w%	Weight percentage
θ	Scattering angle
λ	wavelength of the X-ray

## LIST OF ABBREVIATIONS

$^{13}\text{C}$ -NMR	Carbon nuclear magnetic resonance
$^1\text{H}$ -NMR	Proton nuclear magnetic resonance
5-FU	5-fluorouraci
AGA	Acrylamidoglycolic acid
AIBN	Azoisobutylnitrile
Am	Acrylamide
ATRP	Atom transfer radical polymerization
BAC	N, N'-bis(acryloyl)cystamine
$\text{C}_{11}\text{EO}_{42}$	poly(ethylene oxide)
C20	Cloisite 20
CCD	Central Composite Design
CEC	Cation exchange capacity
$\text{CH}_3\text{I}$	Methyl iodide
CMCS	Carboxymethyl chitosan
CNT-PCs	Carbon nanotube- Polymer Composites
CNTs	Carbon nanotubes
CPT	Camptothecin
Cr(VI)	Chromium primarily
CTAs	Chain-transfer agents
DEAEMA	2-Diethylaminoethyl methacrylate
DEX	Dextran
DHG	Double-hydrophilic glycopolymer
DMAEM	2-Dimethyl aminoethylmethacrylate
DOE	Design of experiment
DOX	Doxorubicin
DSC	Differential scanning calorimetry
DTG	derivative thermal gravimetric
EVOH	Poly(ethylene-co-vinyl alcohol)
FA	Folic acid
$\text{Fe}_3\text{O}_4$ -MWCNTs- COOH	Carboxylic acid functionalized multiwall carbon nanotubes and magnetic iron oxide nanoparticles

Fe <sub>3</sub> O <sub>4</sub>	Magnetite iron oxide
Fe-MWCNTs	Maghemite–multiwalled carbon nanotubes
fib	Fibrinogen
FITC	fluorescein isothiocyanate
FP	Frontal polymerization
FRP	Free radical polymerization
FTIR	Fourier-transform infrared spectroscopy
GC-MS	Gas Chromatography/Mass Spectroscopy
GO	Graphene oxide
HDPE	Polyethylene
HEMA	2-Hydroxyethyl methacrylate
HPCL	Poly(ε-caprolactone)
HPMA	N-(2-hydroxypropyl)methacrylamide
IANa	Itaconic acid sodium
IPTES	3-isocyanato- propyltriethoxysilane
LCST	Low critical solution temperature
LDPE	low-density polyethylene
Meg	Megestrol acetate
MMA	Methyl methacrylate
MMANa	Metacrylic acid sodium
MMT	Montmorillonite
MR	Magnetic resonance
MWCNTs	Multi-walled carbon nanotubes
MWCNTs/PANI/Fe <sub>3</sub> O <sub>4</sub>	MWCNTs/polyaniline/magnetite
MWCNTs-AuNPs	multiwall carbon nanotubes-gold nanoparticles
MWNT-PAmI	MWNT grafted cationic polyelectrolyte
nBMA	n-butyl methacrylate
NGs	Nanogels
NH <sub>2</sub>	Aminopropyl
NMR	Nuclear Magnetic Resonance
NMR	Nuclear magnetic resonance
ONZ	Ornidazole
P(OVNG)	6-O-vinyl-nonanedioyl-D-galactose

P4VP	Poly (4-vinyl pyridine)
PA6	Polyamide 6
PAA	Polyacrylic acid
PAA	Poly(acrylic acid)
PAA	Polyacrylamide
PAMAM-0	Polyamidoamine generation-0
PAN	Polyacrylonitrile
PBD	Plackett-Burman Design
PBMA	Poly(n-butyl methacrylate)
PCL	poly( $\epsilon$ -caprolactone)
PDEAEMA	Poly(2-diethylaminoethyl methacrylate)
PEG	Poly(ethylene glycol)
PEG	poly(ethylene glycol)
PEG	Poly(ethylene glycol)
PEGMA	Poly(ethylene glycol)methacrylate
PEI	Polyethyleneimine
PEI	Polyethyleneimine
PEO	Poly(ethylene oxide)
PEO–Me	Poly(ethylene-oxide) dimethylether
PHPMA	Poly(N-(2-hydroxypropyl)methacrylamide)
PHPMA	Poly(N-(2- hydroxypropyl)methacrylamide)
PLA	Poly lactide
PLA	Poly lactide
PMMA	poly(methyl methacrylate)
PMMA	Polymethyl methacrylate
PNC	polymer nanocomposites
PNIPAM	Poly-(N-isopropylacrylamide)
PNVCL	Poly (N-vinylcaprolactam)
PNVCL–COOH	poly(N-vinylcaprolactam) -Carboxyl-terminated
POE-g-AA	Polyethylene–octene elastomer-grafted metallocene polyethylene–octene elastomer
PP	Polypropylene
PS	polystyrene



PSS	poly(sodium 4-styrenesulfonate)
PtBA-b-PS	Poly [(tert-butyl acrylate)-b-styrene]
PTME	Polytetramethylene ether
PU	Polyurethanes
PUU	Poly(urea urethane)
PVC	Polyvinyl chloride
PVC	poly(vinyl chloride)
PVC	polyvinyl chloride
PVK	Poly(N-vinyl carbazole)
PVOH	Poly(vinyl alcohol)
PVP	poly (4-vinylpyridine)
RAFTP	Reversible addition fragmentation chain transfer polymerization
RhB	Rhodamine B
ROP	Ring opening polymerization
RSM	Response surface methodology
R-Sq	R-squared
R-Sq(adj)	adjusted R-squared
R-sq(pred)	Predicted R-squared
S	styrene
SA	Sodium alginate
SEM	Scanning electron microscopy
SI-ATRP	surface-initiated atom transfer radical polymerization
SR	Swelling ratio
SWCNTs	Single walled Carbon Nanotubes
TEM	Transmission electron microscopy
TGA	Thermogravimetric analysis
THF	Tetrahydrofuran
UA	Undecenoic acid
UCST	Upper critical solution temperature
UV-Vis	Ultraviolet-visible
VCL	vinylcaprolactam

VPTT	Volume phase transition temperature
WPU	Waterborne polyurethane
XRD	X-ray diffraction

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